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(71)Applicant : MERCK PATENT GMBH

(72)Inventor : ANSELMANN RALF

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(54) INORGANIC SPHERICAL ABSORPTION PIGMENT

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain the subject pigment exhibiting high color clearness and uniformity by including silicon dioxide microballoons each having a specific size and a metal oxide.

SOLUTION: This pigment comprises silicon dioxide microballoons each 50 nm to 50 μm in size and coating film containing a metal oxide except titanium dioxide or a metal oxide and a coloring matter; wherein the metal oxide is Fe2O3, FeO(OH), Fe3O4 or Cr2O3, while the coloring matter is azure, carmine red, an organic pigment or organic dye.

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CLAIMS

[Claim(s)]
[Claim 1] The inorganic spherical absorption pigment characterized by becoming the silicon dioxide solid sphere which has a 50-micrometer diameter from 50nm from the coat which contains a metallic oxide and the ** (wearing) color matter, including the metallic oxide except a titanium dioxide.
[Claim 2] The absorption pigment of claim 1 characterized by a metallic oxide being Fe 2O3, FeO(OH), Fe 3O4, or Cr2O3.
[Claim 3] The absorption pigment of claim 1 characterized by the ** (wearing) color matter being Berlin blue, carmine red, an organic pigment, or organic dye.
[Claim 4] Use of one absorption pigment of claims 1-3 for coloring paint, printing ink, plastics, and the cosmetics compound for care and cleaning and an ornament.
[Claim 5] Use based on claim 4 characterized by this compound being a compound of a lipstick, eye shadow, nail enamel, the charge of makeup makeup of a liquid and a solid-state, rouge (red), powder, an emulsion, a lotion, a cream, a sunscreen product, or the OIRI base.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the inorganic absorption pigment which consists of a coat of the spherical silicon dioxide used as a nucleus, and a metallic oxide.
[0002] The inorganic absorption pigment used for the purpose of coloring must be made into the gestalt which brings about the color which can distribute easily first and is reproducible. These conditioning of the pigment which has decisive effect on the quality of a final product, for example, grinding, and ****** start, and cost also starts. When other faults wet wet, they are that the color of a pigment changes. Moreover, with a cosmetics compound, a pigment is a good skin feel. - It is having to have property - which the conventional inorganic absorption pigment's has only in the minimum.
[0003]
[Description of the Prior Art] JP 62-288662 is indicating the yellow pigment which consists of a goethite particle adhering to the nucleus of a silicon dioxide particle, and this. Goethite is alpha-FeO (OH) and this is known as needlelike iron ore again. This yellow pigment is built by grinding the two above-mentioned component by dryness, or mixing and drying continuously in an aquosity alkali solution. Since it separates into that component partially, this pigment has the fault that it is unstable in an application system. This pigment does not fully have a good skin feel for being used as cosmetics.
[0004] Moreover, the color of a pigment lacks in brightness weakly.
[0005] It is also known that a titanium dioxide will cover a spherical silicon dioxide with a wet method. JP 06-011 The spherical silicon dioxide covered with the titanium dioxide is indicated by 872. This pigment is used as a masking reagent in a cosmetics compound.
[0006] WO 94/21 The pigment with the high light-scattering force it is weak from the second layer of the nucleus of a silicon dioxide, the first pass of a titanium dioxide, a silicon dioxide, aluminum silicate, or an aluminum oxide is describing 733.
[0007]
[Problem(s) to be Solved by the Invention] The purpose of this invention is offering the absorption pigment in which the image clarity of a high color and homogeneity are shown, and when it can incorporate into the system which should be colored even if it does not carry out conditioning further and wets wet, even if there is this pigment, only change of merely few colors will be shown.
[0008]
[Means for Solving the Problem] This purpose is reached by the inorganic spherical absorption pigment which serves as a silicon dioxide solid sphere which has a 50-micrometer diameter from 50nm from the coat containing the metallic oxide and/or ** (wearing) color matter except a titanium dioxide according to this invention.
[0009] This purpose distributes further the silicon dioxide solid sphere which has a 50-micrometer diameter from 50nm by 1 to 30% of the weight of concentration in deionized water with the temperature of 50 to 90 degrees C according to this invention. Metal salting in liquid is added at the rate of 0.0001 to 0.1mg as a metal salt per minute per two 1m of silicon dioxide solid spheres by 10 from pH1 5 to 40%.

A base is added to coincidence, pH is kept constant, the silicon dioxide solid sphere covered in this way is separated, the water or the organic solvent which does not have ion completely washes, and it dries at 80 to 150 degrees C, and is attained by the approach of preparing the pigment of this invention by calcinating at 300 to 1,000 degrees C. In other embodiments of the approach of this invention, the ** (wearing) color matter has deposited on it further to the thin layer of a metallic oxide.
[0010] It specifies that this invention uses the pigment of this invention in order to color paint, printing ink, plastics, and the cosmetics compound for care and cleaning and an ornament again.
[0011]
[Embodiment of the Invention] The start raw material used in order to make a pigment is a spherical silicon dioxide which has a 50-micrometer diameter from 50nm. These solid spheres seem to refer to as the approach used in order to build a pigment not having a single path but having a distribution spectrum about the particle size. As for a start raw material, it is useful that 99% of a particle is the fraction whose magnitude is less than 50 micrometers. The fraction whose magnitude 90% of a particle is less than 20 micrometers is desirable.
[0012] Preparation of a spherical silicon dioxide is known. It is built by hydrolyzing organic or an inorganic silicon compound by the emulsion method. Such an approach is DE. 21 55 281 DE 26 10 852 GB 1 141 924 and EP 0 162 It is indicated by 716. A spherical silicon dioxide is called for in a commercial scene. Merck Co. (Merck KGaA) offers the spherical silicon dioxide with the grain size of less than 20 micrometers in the name of RONASUFIA (Ronasphere R).
[0013] an organic pigment, a color, and coming out and covering are performed in a silicon dioxide solid sphere again by the metallic oxide or the ** (wearing) color matter, for example, Berlin blue, carmine red, or the known approach.
[0014] US when covering iron(III) oxide 3 087 828 and US 3087 Like the publication to 829, it is US only from iron(III) salt. 3 874 An iron(II) oxide salt can also be left for 890 like a publication. In this case, the coat of the iron(II) oxide built first oxidizes to oxy-iron(III) hydroxide. It is more desirable to use iron(III) salt as a start raw material. In this case, an iron(III) chloride solution is measured and added into the aqueous suspension of a spherical silicon dioxide by the temperature of 60 to 90 degrees C, and pH of 2.5 to 4.5. pH is kept constant by measuring a sodium-hydroxide water solution to coincidence 32%, and adding to it. This approach is DE. 196 18 It is describing 568.
[0015] The pigment calcinated at 300 to 900 degrees C has Orange thru/or red.
[0016] Although the start raw material used is the mixture of iron(III) sulfate and iron(II) sulfate and this mixture is measured and added by 4.5 from pH2.5 into the 60 to 90-degree C heat suspension of a silicon dioxide solid sphere when covering with the yellow FeO of the form of goethite (OH), pH is kept constant by adding a sodium-hydroxide solution to coincidence 32% in the meantime.
[0017] It is EP to cover with the yellow FeO of the form of goethite (OH) again. 0 It can carry out to 659843 also by the approach of a publication. In this case, FeSO4 water solution of pH1.5 is measured in the 70-degree C heat suspension of a spherical silicon dioxide in nitrogen-gas-atmosphere mind, and using Na2CO3 solution, pH is adjusted to 4 and, in addition, is continuously kept constant by pH4 again.

[0018] By both of the approaches, a golden pigment is obtained, and this pigment is processed by the conventional approach and dried at 80 to 130 degrees C.
[0019] In order to obtain a black pigment, the solid sphere of the silicon dioxide covered with FeO (OH) is heated at 300 to 1,000 degrees C in a hydrogen air current or activation gas (N2/H2 of various rates), and produces a black iron oxide (II, III).
[0020] Green pigments cover the solid sphere of a silicon dioxide with chrome oxide, and are obtained. Covering is performed by being 60 to 90 degrees C, and measuring and adding a chromium chloride solution into the heat suspension of the silicon dioxide solid sphere of 9 from pH5.5. A pigment is heat-treated at 500 to 1,000 degrees C after the usual processing. The obtained pigment is green.
[0021] If ferrocyanicacid iron (III) is settled on a silicon dioxide solid sphere, the pigment of dark blue will be obtained. For that, an iron(III) sulfate water solution and a potassium-hexacyanoferrate(II) water solution are measured to coincidence, and are put in by 4 from pH1.8 into the 60 to 90-degree C heat

dispersion liquid of a spherical silicon dioxide. This pigment is processed by the conventional approach and it dries at 80 to 130 degrees C.
[0022] It is advantageous, if 5 thru/or 20% of the weight of a titanium dioxide are made to sediment on a silicon dioxide first to paste up a Berlin-blue coat on a spherical silicon dioxide well and a front face is activated.
[0023] Covering by the titanium dioxide is US. 3 553 001 and EP 0 803 It is carried out to 550 by the approach of a publication. About 0.5 to 5 and pH especially fixed on the real target of about 1.5 to 2.5 are maintained by measuring a base, for example, aqueous ammonia, solution or hydroxylation alkali-metal water solution to coincidence in the meantime, and adding, although 100 degrees C of titanium salt water solutions are especially added to the heat suspension of a 70 to 80-degree C spherical silicon dioxide gradually from 50. If the thickness of a desired layer is obtained by TiO2 sediment, addition of titanium salting in liquid will be stopped immediately.
[0024] This approach learned also as a dropping test avoids the excess of a titanium salt. This is reached when the effective-surface product of the particle which is required to cover uniformly and should be covered with hydration TiO2 supplies the hydrolyte of only the amount per [absorbable] unit time amount to per unit time amount. Therefore, there is no generation of the hydration titanium-dioxide particle which does not deposit on the front face which should be covered. The amount of the titanium salt added in 1 minute by this approach is 2xten - four [about 0.01 to] mols in extent as a titanium salt per square rice of the surface area which should be covered.
[0025] The silicon dioxide solid sphere covered only with the titanium dioxide is useful also as an absorption pigment.
[0026] The covered metallic-oxide content of a silicon dioxide solid sphere is in 1 to 80% of the weight of the range.
[0027] Although the absorption pigment of this invention is used in order to color paint, printing ink, and plastics, it is especially used into the cosmetics compound for care and cleaning and an ornament. There is a compound of a lipstick, eye shadow, nail enamel, the charge of makeup makeup of a liquid and a solid-state, rouge (red), all kinds of powder, an emulsion, a lotion, a cream, a sunscreen product, and the OIRI base in these.
[0028] A pigment is contained by 0.1 to 80% of the weight of concentration in these compounds.
[0029]
[Example] It distributes in the 1,900g water which does not have ion completely, stirring silicon dioxide solid sphere [RONASUFIA (Ronasphere R) of 1100g of examples which explains this invention according to the following examples, and Merck Co. (Merck KGaA)], and heats at 75 degrees C, and a hydrochloric acid adjusts to pH2.7 10%. Next, 15% iron(III) chloride solution is measured and put in, adding a sodium-hydroxide water solution to coincidence 32%, and keeping pH constant. For [of the beginning] 20 minutes, the addition rate of an iron(III) chloride solution is a part for 2.4ml/until a 45-degree hue angle is acquired for [of 0.7ml a part for /and a degree] 20 minutes after [2.4ml] a part for /, and 40 minute. A hue angle is measured using Minolta CR-300 colorimeter. Next, suspension is stirred for 30 more minutes and pH is adjusted to 5 using a sodium-hydroxide water solution 32% after it. A solid-state is separated on a suction filter, it washes until a salt is lost with the water which does not have ion completely, and it dries at 110 degrees C, it calcinates for 30 minutes at 800 degrees C, and the fine particles of the dark reddish-brown whose iron(III) oxide content is 55% are obtained.
[0030] It distributes in the 2,277g water which does not have ion completely, stirring the silicon dioxide solid sphere of 2120g of examples, heats at 80 degrees C, and adjusts to pH3.2 using a sulfuric acid 20%. Next, it adds, measuring the water solution which melted 338g iron(III) sulfate and 46.4g iron(II) sulfate 7 hydrate in 1,000g water. A sodium-hydroxide water solution is added to coincidence 32%, and pH is kept constant. An addition rate is a part for 1.9ml/1.3ml for [as follows] 10 minutes further for [of 0.6ml a part for /and a degree] 10 minutes for [of the beginning] 10 minutes. After 30 minute, an addition rate is gathered to a part for 2.6ml/, and addition of an iron-sulfate solution is continued until a 65-degree hue angle is measured. Next, pH is adjusted to 7.0 using a sodium-hydroxide water solution 32%. Next, this pigment is processed like a publication in the example 1, it dries at 110 degrees C, and

the golden color fine particles whose content of an iron oxide is 50 % of the weight are obtained.
[0031] 10g of yellow pigments adjusted according to example 3 example 2 is paid to the glass boat made from a quartz, and it moves into the tube made from a quartz. It moves for 30 minutes into the furnace by which carried out the flash plate of this tube for 15 minutes by activation gas (hydrogen), and the preheating was carried out at 550 degrees C. Next, a tube is taken out from a furnace, and it cools, exposing to activation gas further. The obtained pigment contains 50% of iron oxide (II, III) (Fe 3O4).
[0032] It distributes stirring the silicon dioxide solid sphere of 450g of examples in the 950g water which does not have ion completely, heats at 75 degrees C, and adjusts to pH6.0. Next, a sodium-hydroxide water solution is added to coincidence 32%, and it adds, measuring the water solution which melted chromium chloride 6 303.8g hydrate in the 777.8g water which does not have ion completely while keeping pH constant. The rate of addition is a part for 1.25ml/for [of the beginning] 30 minutes. A rate is gathered to a part for 2.5ml/until it finishes carrying out measuring addition of the solution of a chromium chloride continuously. It stirs for 30 more minutes. Next, the deep green fine particles which process like a publication in the example 1, dry at 110 degrees C, calcinate this pigment for 30 minutes at 800 degrees C, and have the tint of thin blue are obtained. The content of chrome oxide is 63.6%.
[0033] It distributes stirring the silicon dioxide solid sphere of 5120g of examples in the 2,280g water which does not have ion completely, and heats at 75 degrees C, and a hydrochloric acid adjusts to pH2.0 10%. Next, it adds, measuring the mixture of a titanium-tetrachloride solution and 83.2g water 60 94.9g%, adding a sodium-hydroxide water solution to coincidence 32%, and keeping pH constant. The rate of addition is a part for 2.2ml/henceforth in a part for 1.0ml/for [of the beginning] 20 minutes. If addition of a titanium-tetrachloride solution finishes, mixture will be stirred for 30 more minutes. Next, it adds, making only into coincidence the solution which melted 57.5g iron(III) sulfate in 575g water while adding the sulfuric acid to coincidence 20% and keeping pH constant, and the solution which melted 74.9g potassium hexacyanoferrate(II) in 575g water, and measuring it separately. The rate of addition is a part for 2.0ml/for [of 0.5ml a part for /and a degree] 20 minutes for [of the beginning] 20 minutes after [1.0ml] a part for /, and 40 minute. If addition of these two solutions finishes, mixture will be stirred for 30 more minutes. A pigment is processed like a publication in the example 1, it dries at 110 degrees C, and the blue fine particles whose Berlin-blue content is 37 % of the weight are obtained.
[0034] It distributes stirring the silicon dioxide solid sphere of 690g of examples in the 1,710g water which does not have ion completely, this suspension is heated at 75 degrees C, and a hydrochloric acid adjusts to pH2.2 10%. It adds into suspension, measuring the solution built with 61.8 520.7g% titanium-tetrachloride solution and 552g water while adding the sodium-hydroxide water solution to coincidence 32% and keeping pH constant. For, it is a part for 1.2ml/, and it continues for [of the beginning] 60 minutes and the rate of addition is a part for 2.6ml/. If addition of a titanium-tetrachloride solution finishes, mixture will be stirred for 30 more minutes and then a hydrochloric acid will adjust it to pH7.0 10%. Next, it adds, making only into coincidence the specific silicate solution built with the solution which melted the 9.39g zinc chloride in 220g water, the specific silicate solution in 220g of water, 90.3g sodium water glass (2 27 % of the weight of SiO(s)), and 233g water, and measuring it the rate for 2.5ml/separately. Mixture is continuously stirred for 30 more minutes. A pigment is processed like a publication in the example 1, and the white fine particles which dry at 110 degrees C, calcinate for 30 minutes at 600 degrees C, and consist of 35.5 % of the weight of silicon dioxide solid spheres, 53 % of the weight of titanium dioxides, 2 % of the weight of zinc oxides, and 9.5 % of the weight of silicon dioxides are obtained.
[0035]
Example 7 Coloring day cream A SAP white (SiO2 solid sphere covered with TiO2, ZnO, and SiO2) 4.00% SAP yellow (SiO2 solid sphere covered with FeO (OH)) 2.00% SAP Orange (SiO 2 solid sphere covered with Fe 2O3) 0.20% SAP red (SiO 2 solid sphere covered with Fe 2O3) 0.20% 0.20% (SiO 2 solid sphere covered with Fe 3O4) of SAP black KARION F liquid (sorbitol) 5.00% Allantoin 0.50% Keltrol T (xanthan gum) 0.20% KEMAGU 2000 (imidazolidinyl urea) 0.30% You KISHIRU K400 (methyl BUROMOGURU taro nitril, phenoxyethanol) 0.10%4-oxy-methyl benzoate (methylparaben)

0.15% Deionized water ** 100.00%B ARASERU 165 (a stearin acid glycerol, PEG- 100 stearate) 2.50% MONTANOBU 68 (SETEA reel alcohol, SETEARIRUGU RIKOSHIDO) 2.50% Dow Corning 345 (cyclomethicone) 1.00% Cetiol SN (iso nonoic acid SETEA reel) 5.00% YUTA Norian G (octyl dodecanol) 4.00% Xia fat (BUCHIROSUPERUMAMU par key) 4.00% 4-oxy-benzoic-acid propyl (propylparaben) 0.05%C Perfumed oil bouquet PUDORE 0.10% preparation: Keltrol It removes and all the components of a phase A are distributed in water. Stirring, Keltrol is sprinkled and it heats at 80 degrees C after 15 minutes. A phase B is heated at 75 degrees C. Stirring gently, a phase A is put in into a phase B and it homogenizes. It cools applying and stirring a phase C at 40 degrees C.
[0036]
Example 8 Lipstick A SAP red (SiO 2 solid sphere covered with Fe 2O3) 10.00% SAP white (SiO2 solid sphere covered with TiO2, ZnO, and SiO2) 5.00%B Beeswax 8.75% RUNASERA C44 (ceresin) 5.25% ADEPUSURANAE SP(lanolin)3.50% Myristic-acid isopropyl 5.60% Liquefied paraffin 2.10% Oxy-NEKUSU K liquid (PEG-8, tocopherol, and palmitic-acid ASUKORUBIRU, an ascorbic acid, citric acid) 0.05% 4-oxy-benzoic-acid propyl (propylparaben) 0.10% Castor oil 100.00% of ** C Tongue dress (perfume) 0.20% preparation: Component of a phase B It heats and fuses at 75 degrees C. It stirs until it adds these pigments to a phase A and becomes homogeneity. This lip stick material is stirred for 15 minutes in the heated 65-degree C casting equipment next, and perfume is paid. the casting which carried out the preheating of the homogeneous melt to 55 degrees C -- public funds -- it slushes into a mold. Next, metal mold is cooled and it takes out after cooling a casting article. After warming this lip stick to a room temperature, it hits against short-time flame.
[0037]
Example 9 Rouge (red)
A SAP red (SiO 2 solid sphere covered with Fe 2O3) 21.50% SAP white (SiO2 solid sphere covered with TiO2, ZnO, and SiO2) 7.00% SAP Orange (SiO 2 solid sphere covered with Fe 2O3) 0.50% SAP yellow (SiO2 solid sphere covered with FeO (OH)) 0.50% SAP black (SiO 2 solid sphere covered with Fe 3O4) 0.50% Mica 10.00% Talc 18.00% Kaolin (kaolin) 25.00% Amylum oryzae 5.00% Magnesium stearate 2.00%B Binder: Myristic-acid isopropyl 8.00% 1.00% (dimethicone, dimeticonol) of Dow Corning 1403 liquid Dow Corning 200 (350cs) liquid (dimethicone) 1.00% preparation: Component of a phase A In addition, it mixes beforehand and applies to a screen (100 micrometers). Next, a binder is dropped, stirring. These fine particles are pressurized by 40 to 50 bars.
[0038]
Example 10 Eye shadow A SAP blue (SiO 2 solid sphere covered with TiO2 and Berlin blue) 10.00% SAP green (SiO2 solid sphere covered with Cr 2O3) 7.00% SAP white (SiO2 solid sphere covered with TiO2, ZnO, and SiO2) 13.00% Mica 10.00% Talc 17.00% Kaolin (kaolin) 24.50% Amylum oryzae 2.50% Magnesium stearate 2.00% Aerosil 200 (silica) 5.00%B Binder: Myristic-acid isopropyl 8.00% Dow Corning Q2 -1403 liquid (dimethicone, dimeticonol) 1.00% Dow Corning 200 (350cs) liquid (dimethicone) 1.00% preparation: Component of a phase A In addition, it mixes beforehand and applies to a screen (100 micrometers). Next, it mixes, dropping and stirring a binder to this fine-particles blend. These fine particles are pressurized by 40 to 50 bars.
[0039]
[Effect of the Invention] The pigment of this invention has many advantages compared with the conventional inorganic absorption pigment. This pigment can be made to mix in an oil phase or the aqueous phase by stirring. It is not necessary to grind beforehand. Regardless of any pretreatments, a pigment fully discovers the tinting strength immediately. consequently, the color which a compound is expected -- reproducible -- the case of the oxide pigment of - former -- like - it is not necessary to collate once again, and if , it is adjusted after grinding. Since the color of a metallic-oxide pigment becomes settled mainly according to the magnitude of a particle, and distribution, a semi- mono dispersion deposit of the metallic-oxide particle to a silicon dioxide particle top brings about the image clarity of a very good color, and the homogeneity of a color.
[0040] The skin feel of the pigment of this invention is excellent on account of a spherical form and spherical magnitude. The metallic oxide of this invention is very smooth to the conventional metallic-

oxide pigment sensing coarse coarsely, and it seems to be velvet.
[0041] When the pigment of this invention is damp compared with the conventional metallic-oxide pigment, there are few degrees of discoloration clearly.
[0042] The mixture of a silicon dioxide solid sphere and the conventional metallic-oxide pigment shows few remarkable weak colors of brightness in an application system rather than the pigment of this invention.
[0043]
[Table 1]

本発明の顔料と、二酸化珪素球体と有色顔料の混合物との色比較

赤

顔　料	L	a	b	h°	C
50%SiO_2球体+50%Fe_2O_3	47.95	24.01	16.36	34.2	29.05
発明の顔料（Fe_2O_3で被覆したSiO_2球体）	44.70	25.79	18.12	35.1	31.51

黄

顔　料	L	a	b	h°	C
50%SiO_2球体+50%FeO(OH)	69.99	7.60	41.44	79.7	43.13
発明の顔料（FeO(OH)で被覆したSiO_2球体）	65.58	13.10	46.99	74.5	48.78

白

顔　料	L	a	b	h°	C
50%SiO_2球体+50%TiO_2	92.68	−0.60	0.47	142.0	0.76
発明の顔料（TiO_2で被覆したSiO_2球体）	96.24	−0.50	1.74	105.9	1.81

黒

顔　料	L	a	b	h°	C
50%SiO_2球体+50%Fe_3O_4	35.76	0.78	−0.32	337.8	0.84
発明の顔料（Fe_3O_4で被覆したSiO_2球体）	33.41	0.40	1.47	74.9	1.52

h° ： 色相角

C ： 彩度

[0044] When a color is compared, it turns out that the pigment of this invention has saturation clearly higher than the mixture of a silicon dioxide solid sphere and the conventional metallic-oxide pigment. Furthermore, the white pigment of a thing with the black colors of this invention darker (low L value) bright (high L value) again of this invention is clearer from L value of white and black. Although dealt in the same effectiveness in an application system as a result, there are few pigments of this invention, and it lives in it.

[Translation done.]